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A PILOT PLANT FOR PRODUCING A MIXTURE OF CRUDE DICARBOXYLIC ACIDS FROM SHALE

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by

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In the laboratories of the Lenirgrad Technological Institute (LTI) imeni Lensovet studies have been conducted investigating the oxidation of shale flotation concentrate in an aqueous-alkali medium by air oxygen [1].

Results of these studies have led to the conclusion that it is practical and feasible to use shale as a raw material for producing dicarboxylic acids.

Further works led to the creation of a design lay-out for this process [2].

However, there is inadequate laboratory data for constructing large-scale production of dicarboxylic acids from shale.

In connection with this a batch process pilot plant was constructed at the "Slantsy" shale-processing combine in the town of Slantsy; the main purpose of the unit is analysis of technological parameters of the process and testing operational effectiveness of new equipment. In addition, the production of significant amounts of acids in this plant will make it possible to conduct extensive studies concerning their application in industry.

The illustration shows a flow diagram of the production of a mixture of crude dicarboxylic acids.

A suspension containing shale flotation concentrate (SFC) and alkali enters jacket-tube gas-lift reactor 5 [3]. Air is supplied under 40 atm pressure. The reactor is heated by steam (temperature about 230°) and cooled by water, both fed into the space between the tubes. The reactor has 20 bubbling and 17 circulating pipes 4 m long. The net volume of the reactor is 250 l. The jacket-tube gas-lift reactor is used for the oxidation of shale because it provides an extended surface of phase contact, intensive circulation of the suspension preventing settling of solid particles along the bottom of the reactor which promotes heat and mass transfer and even distribution of ingredients throughout the reactor.

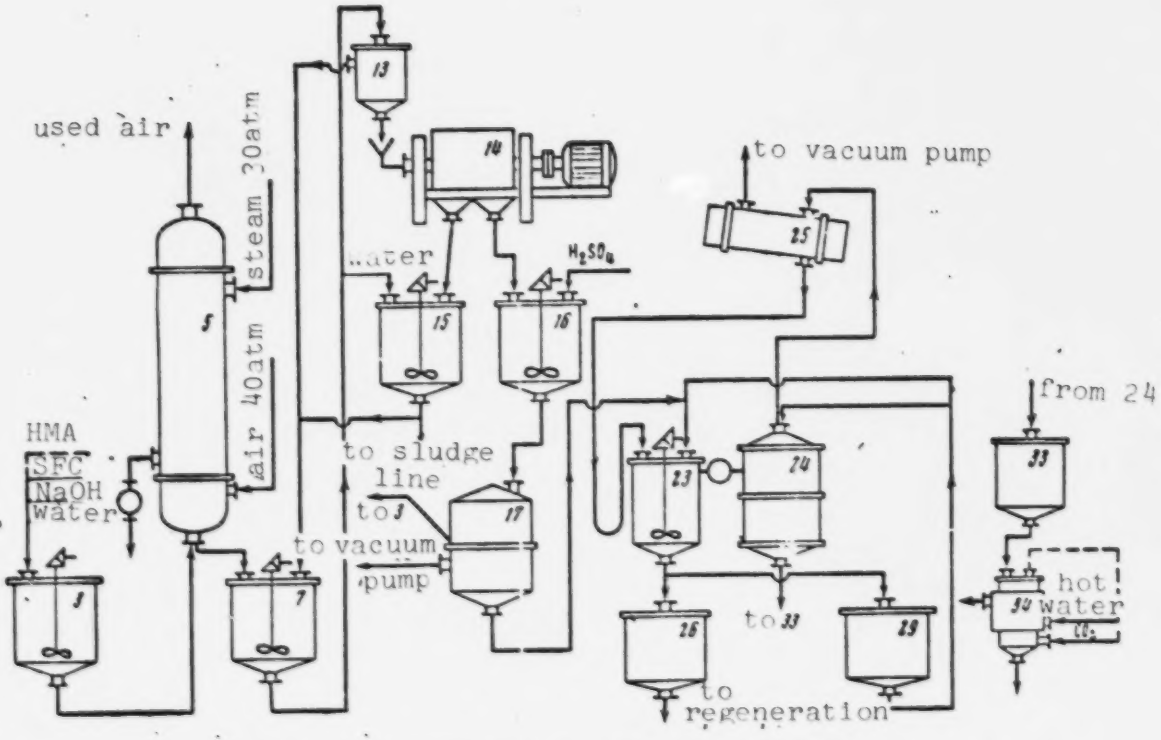

Схема производства смеси сырых дикарбоновых кислот

Flow diagram of the production of a mixture of crude dicarboxylic acids

The reactor lacks mechanical mixing equipment, but the production process is analogous to that of ordinary jacket-tube heat exchangers.

After oxidation the cooled suspension is directed to a NOGShch-315H centrifuge 14 for separation of unoxidized shale and ash. The residuum is dumped into the department sludge line and the separated solution together with the washing water enters neutralizer 16 which is equipped with a propeller mixer. Concentrated sulfuric acid is supplied here. The rather long neutralization time requires the installation of three successive units. The high molecular acids (HMA) precipitated during neutralization are separated in vacuum filter 17. Beyond the filter HMA are returned to tank 3 and again exposed to oxidation, making it possible to increase the yield of diacids.

Diacids are extracted from the aqueous solution in a batch process extraction unit composed of extractor 23, evaporator 24, condenser 25, operating under discharge. The extractor has a propeller mixer and a ring settling zone. The evaporator is in the form of an evaporating unit with an interior heating chamber where hot water is fed into the space between the tubes. When the evaporator is heated, ethyl acetate vapors enter the condenser and then the mixing zone of the extractor. From the mixing zone the ethyl acetate solution of dicarboxylic acids enters the settling zone and then by gravity reaches the evaporator. Thus, there is continuous circulation of ethyl acetate. Upon completion of extraction the aqueous-saline solution from the extractor is regenerated to distill dissolved ethyl acetate. In the evaporator the extractant is distilled and the mixture of extracted dicarboxylic acids concentrated by evaporation.

From the evaporator the mixture of crude dicarboxylic acids enters dryer 34. The dryer is a cylindrical vessel with a jacket and a false perforated bottom under which is fed carbon dioxide heated to 70°. Final removal of ethyl acetate occurs in bubbling through the layer of dicarboxylic acids. Tests have shown that the technological parameters of the oxidation of shale kerogen in an aqueous-alkali medium with air oxygen in a pilot plant are close to optimum parameters of the process obtained in a laboratory enlarged plant.

The mixture of crude dicarboxylic acids produced in a pilot plant can be used without separation only for the synthesis of rigid polyurethane foam plastics, which is one drawback of this plan. Therefore, the extraction of dicarboxylic acids from an oxidate with ethyl acetate is now replaced by the sorption method of extracting and separating dicarboxylic acids, devised at the LTI imeni Lensovet. The use of this method makes it possible to produce not only narrow fractions of dicarboxylic acids, but also individual acids, expanding the area of use of "shale" acids sharply and markedly improving economic indices of the entire process.

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